AD-769 742

SOME NEW POTENTIAL CERAMIC-METAL ARMOR MATERIALS FABRICATED BY LIQUID METAL INFILTRATION

Michael W. Lindley, et al

Army Materials and Mechanics Research Center
Watertown, Massachusetts

September 1973

DISTRIBUTED BY:

NTIS

National Technical Information Service
U. S. DEPARTMENT OF COMMERCE
5285 Port Royal Road, Springfield Va. 22151

The findings in this report are not to be construed as an official Department of the Army position, unless so designated by other authorized documents.

Mention of any trade names or manufacturers in this report shall not be construed as advertising nor as an official indorsement or approval of such products or companies by the United States Government.

ib

DISPOSITION INSTRUCTIONS

Destroy this report when it is no longer needed.
Do not return it to the originator.

UNCLASSIFIED
Security Classification

AD 769 742

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Army Materials and Mechanics Research Center Watertown, Massachusetts 02172	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP

3. REPORT TITLE
SOME NEW POTENTIAL CERAMIC-METAL ARMOR MATERIALS FABRICATED BY LIQUID METAL INFILTRATION

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

5. AUTHOR(S) (First name, middle initial, last name)
Michael W. Lindley and George E. Gazza

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
September 1973	20	9

8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S)
b. PROJECT NO. D/A 1T062105A328	AMMRC TR 73-39
c. AMCMS Code 612105.11.294	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)
d. Agency Accession Number DA OE4692	

10. DISTRIBUTION STATEMENT
Approved for public release; distribution unlimited.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
	U. S. Army Materiel Command Alexandria, Virginia 22304

13. ABSTRACT
An investigation was initiated to explore economical methods of fabricating potential lightweight ceramic armor materials based on liquid metal infiltration processes. Fabrication, microstructural, and limited mechanical property data on AlB_{12}, SiB_6, B_4C, boron, and various mixtures, infiltrated with liquid silicon and aluminum are presented. The possibility of introducing limited ductility in such ceramic-metal systems and the concept of a "gradient" armor are considered, and successful preliminary experiments are presented. (Authors)

ia

DD FORM 1473 REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

UNCLASSIFIED

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Ceramic composites						
Fluid infiltration						
Silicon						
Aluminum						
Metallography						
Borides						
Carbides						
Bending stress						

ic

AMMRC TR 73-39

SOME NEW POTENTIAL CERAMIC-METAL ARMOR MATERIALS FABRICATED BY LIQUID METAL INFILTRATION

Technical Report by

MICHAEL W. LINDLEY and GEORGE E. GAZZA*

September 1973

D/A Project 1T062105A328
AMCMS Code 612105.11.294
Metals Research for Army Materiel
Agency Accession Number DA OE4692

*Senior Scientific Officer, Admiralty Materials Laboratory, Poole, Dorset, United Kingdom.

CERAMICS RESEARCH DIVISION
ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

AMMRC Ex. O. 33A48

id

ARMY MATERIALS AND MECHANICS RESEARCH CENTER

SOME NEW POTENTIAL CERAMIC-METAL ARMOR MATERIALS FABRICATED BY LIQUID METAL INFILTRATION

ABSTRACT

An investigation was initiated to explore economical methods of fabricating potential lightweight ceramic armor materials based on liquid metal infiltration processes. Fabrication, microstructural, and limited mechanical property data on AlB_{12}, SiB_6, B_4C, boron, and various mixtures, infiltrated with liquid silicon and aluminum are presented. The possibility of introducing limited ductility in such ceramic-metal systems and the concept of a "gradient" armor are considered, and successful preliminary experiments are presented.

CONTENTS

Page

ABSTRACT

INTRODUCTION 1

SYSTEMS INVESTIGATED 1

EXPERIMENTAL PROCEDURE

Preparation of Specimens 2

Crucibles and Furnacing 3

Silicon Metal Infiltration 3

Aluminum Metal Infiltration 3

Characterization of Materials 3

SINGLE INFILTRATION RESULTS

Single Infiltration Experiments with Silicon 4

Single Infiltration Experiments with Aluminum 6

MECHANICAL PROPERTY RESULTS AND DISCUSSIONS OF SINGLE INFILTRATION STUDIES 8

DISCUSSION AND DUAL INFILTRATION RESULTS 10

Gradient Ductile/Brittle Material Based on a Single Component Ceramic - The Effect of Particle Size 13

Gradient Ductile/Brittle "Ceramic" Based on a Two-Component Ceramic - The Effect of Wettability 14

SUMMARY 15

ACKNOWLEDGMENTS 15

LITERATURE CITED 16

INTRODUCTION

In recent years, low density, high elastic modulus, high hardness ceramics have been utilized extensively for lightweight armor applications by the U. S. Armed Forces and to a very limited extent by the U. K. Armed Forces. Considerable sums of money have been expended to produce ceramic armor products that, by the very nature of their component parts and more particularly by their method of fabrication (such as hot pressing), are expensive. A state-of-the-art review of ceramic lightweight armor materials has recently appeared.[1] One less expensive ceramic armor material (cheaper than an equivalent hot-pressed material) is based on the infiltration of silicon into a boron carbide/carbon preform; this material has been developed commercially and is being utilized by the U. S. Armed Forces.[1]

Additionally, it has been persuasively argued that the introduction of a limited amount of ductility[2] (up to 0.5% strain or more) at the rear face of a composite ceramic-reinforced plastic armor system could have a significant effect in increasing the ballistic limit of a ceramic armor system. Wilkins[2] pictures essentially a uniform two component system of a ceramic and a metal component and also a graded system with a "graded cermet" transition zone changing smoothly from 100% ceramic at the ceramic side of the zone to 100% metal at the metal side of the zone. Such a system would be able to sustain the reflected tensile wave (resulting from ballistic impact) longer before ultimate failure by fracture than a system with essentially zero ductility. Various approaches to this problem apart from liquid metal infiltration have been advocated including the hot-pressed Al_2O_3-Mo gradient system,[3] however, liquid metal infiltration would seem to have the great attraction of relatively low temperature processing compared to hot-pressing processes.

A previous study on the fabrication of graded ceramic-metal systems by infiltration was reported by Goetzel and Adamec[4] in 1956. Nickel alloys were infiltrated into cold-pressed titanium carbide bodies which had been formed by using pressure gradients and selective particle sizes. Their primary interest was to produce materials for use in high temperature engine components such as turbine buckets and nozzle vanes.

With these criteria and background in mind an experimental survey was mounted on liquid metal-infiltrated ceramic armor materials: low density and immediate availability in powder form were selection criteria for the ceramic components, and low melting point and low density were the economic and density conditions relevant to the liquid metal infiltrants.

SYSTEMS INVESTIGATED

Potential lightweight ceramic armor materials immediately available in powder form were B_4C, alpha-AlB_{12}, SiB_6, SiC, alpha-Al_2O_3, TiB_2, CaB_6, and boron. Metal and metalloid infiltrants considered were aluminum (melting point 660 C, density 2.70 g/cc), titanium (melting point 1675 C, density 4.54 g/cc), and silicon (melting point 1418 C, density 2.33 g/cc). Nickel was eliminated due to its high density (8.9 g/cc). Titanium was finally rejected due partly to its density and partly because liquid infiltration temperatures would be similar to temperatures used in conventional hot-pressing processes and hence processing costs would still be relatively expensive. The systems investigated are shown in Table I.

Table I. MATERIAL COMPONENTS USED FOR INFILTRATION STUDIES

Single Infiltration Studies		Dual Infiltration Studies
Silicon Infiltrant	Aluminum Infiltrant	Silicon and Aluminum Infiltrants in a Two-Stage Process
100% Boron 100% AlB_{12} 100% B_4C (BPI) 100% B_4C (NC) 90% B_4C (NC)/10% Boron 80% B_4C (NC)/20% Boron 100% SiB_6 80% SiB_6/20% Boron	100% Boron 100% SiB_6 100% AlB_{12} 80% AlB_{12}/20% Boron 60% AlB_{12}/40% Boron 100% B_4C (NC)	AlB_{12} B_4C-Si/SiB_6-Al AlB_{12}-Si/SiB_6-Al

Notes:

1. Boron: An amorphous grade produced by AREMO Products Inc., Briarcliff Manor, New York. Aluminum and silicon infiltrants were standard laboratory grade powders from Fisher Scientific Co., Fairlawn, New Jersey.

2. AlB_{12}: -320 mesh produced by the Norton Company, Worcester, Mass.

3. B_4C (BPI): "Hot pressing grade" B_4C produced by Boride Products Inc., Traverse City, Michigan.

4. B_4C (NC): -320 mesh (Norbide), produced by the Norton Company, Worcester, Mass.

5. SiB_6: -320 mesh produced by Cerac Company, Butler, Wisconsin.

EXPERIMENTAL PROCEDURE

Preparation of Specimens

Specimens were cold pressed in one-inch-diameter double-action hardened steel dies at 17,700 psi (123 $MN.m^{-2}$). Dry powders were used to produce compacts approximately 0.3 inch (8.5 mm) thick by 1.0 inch (25.4 mm) diameter. The green densities of pressed compacts were determined from their final dimension and weight of powder and were typically 60% to 70% of theoretical density. Compacts were too fragile for water immersion or mercury immersion density determinations. In early experiments cold-pressing pressures of 11,200 psi (80 $MN.m^{-2}$) and 6,700 psi (46 $MN.m^{-2}$) were used but no conclusive effect on the infiltration process could be seen on the few samples examined and at these lower pressures the boron carbide compacts were very fragile. In some pressings, a 4 w/o solution of Carbowax was added which was subsequently removed on firing, but with the particle size range of the materials examined there appeared no particular advantage and and in most cases inferior infiltrated materials resulted.

Crucibles and Furnacing

The single closed-end crucibles used to contain the liquid metal infiltrant on the specimen were machined from graphite to 1-1/4 inches internal diameter (1-1/2 inches outside diameter) and 1-1/4 inches or 2 inches deep. For aluminum infiltration, the crucible was lined with 0.010-inch-thick "Grafoil" graphite sheet (Metallurgical Grade GTB)* which facilitated removal of the infiltrated body and eliminated reaction with the graphite crucible. When silicon was the infiltrant, a layer of boron nitride was used between the specimen and the crucible to avoid reaction of the infiltrated specimen and the crucible material. The compact to be infiltrated was placed on top of the required amount of powdered or granular metal in the bottom of the crucible; in some cases the powdered infiltrated metal was also placed on top of the pressed compact, and in some cases a Grafoil lid to the crucible was used.

All firing schedules were in a tungsten resistance-heated vacuum furnace operating at reduced pressure with an argon atmosphere; temperatures were measured by a W/Rh thermocouple system.

Silicon Metal Infiltration

Typical heat treatment schedules were 15 hours evacuation at 10^{-5} torr at room temperature followed by outgassing at 10^{-5} torr and 750 C for 2 hours, typical heating rates from room temperature to 750 C being 20 C per minute. Commercially pure argon (oxygen level unknown) was bled into the system at 1000 μm mercury pressure and the temperature was raised to 1420 C at 30 C per minute and finally to 1500 C for a hold of 2 hours. The compacts were slow cooled through the silicon solidification temperature (1418 C) at 50 C per hour and then cooled from 1400 C at 10 C per minute.

Aluminum Metal Infiltration

Typical heat treatment schedules were 15 hours evacuation at 10^{-5} torr at room temperature followed by 1-1/2 hours at 10^{-5} torr at 500 C, heating rates to 500 C being 20 C per minute. Initial attempts at infiltrating AlB_{12} at 750 C (melting point of aluminum is 670 C) were unsuccessful, but increasing the infiltration temperature to 1100 C with a hold of 2 hours resulted in complete infiltration (an argon pressure of 1000 μm of mercury was maintained from 500 C to 1100 C). Heating rates from 500 C to 1100 C were typically 20 C per minute as were cooling rates from 1100 C to 700 C. Compacts were slow cooled through the solidification temperature for aluminum and cooling rates from 650 C were typically 5 C to 10 C per minute.

Characterization of Materials

Infiltrated materials were examined as mounted polished sections by light microscopy, and by X-ray diffractometer diffraction methods using CuKα radiation. Densities were determined on some materials by standard water immersion techniques, although porous specimens were first coated with a sprayed coating of Krylon acrylic spray.

*Union Carbide Corporation, Carbon Prod. Div.

Modulus of rupture and elastic modulus data, where measured, were initially determined using 4-point bend test specimens with an outer span of 15.25 mm (0.6 inch) and an inner span ot 6.35 mm (0.25 inch) on an Instron Testing Machine. Specimen dimensions were 20.3 mm (0.8 inch) × 3.05 mm × 3.05 mm (0.12 inch) and specimens were tested in the surface diamond ground condition (no special surface finishing treatment). Bend bars were also machined from 2-inch-diameter infiltration specimens and tested in 4-point bending using an outer span of 40.64 mm (1.60 inch) and an inner span of 12.70 mm (0.5 inch). Specimen dimensions were 50.8 mm (2.0 inch) × 6.35 mm × 6.35 mm (0.25 inch). Elastic modulus values were determined from the strain gage (SR-4) instrumented tensile faces of the modulus of rupture bars.

Fracture surfaces of test bars were examined by scanning electron microscopy (AMR machine), the specimens being gold coated for electrical conduction.

The samples were examined by electron probe microanalysis at Advanced Metals Research, Burlington, and the Admiralty Materials Laboratory. Both machines were equipped with a light element analysis facility for detecting boron.

Microhardness values, where measured, were recorded on a Wilson Tukon Tester, Knoop values being recorded with a 100-gram applied load, the mean of eight readings being determined.

SINGLE INFILTRATION RESULTS

Single Infiltration Experiments with Silicon

1. SiB_6-Si and 80% SiB_6/20% boron-Si. There was no evidence of infiltration in these systems under the conditions employed, however, a thin 150-μm skin was observed on the SiB_6-Si specimens.

2. Boron-Silicon. Incomplete penetration to a depth of 1 to 2 mm observed, the compact was very friable at the center.

3. AlB_{12}-Si. Complete infiltration observed in this system. The rather complex microstructure of this material in the as-polished condition is shown in Figure 1. Electron probe microanalysis showed the white interconnecting boundary network to be essentially 100% silicon but with approximately 0.3 w/o aluminum and less than 0.2 w/o boron as other constituents. The rectangular dark grey phases within this silicon area were identified as probably alumina (48 w/o measured aluminum content). The light grey matrix, as expected, was largely a complex AlB_{12} containing small amounts of silicon while other areas were probably SiB_6 containing small amounts of aluminum (5 w/o). The white metallic inclusions within these complex Si-B-Al areas were shown to be essentially Fe/Si/Al regions of approximately equal proportions, possibly resulting from iron-combining impurities in the starting materials (the silicon granules were 97% pure; iron is an impurity usually associated with silicon).

A density of 2.4 g/cc was measured; a 100% dense 60 w/o AlB_{12}/40 w/o silicon compound would theoretically have a density of 2.50 g/cc. In the absence of a total silicon content is is not possible to calculate theoretical density for this material.

The X-ray diffraction patterns of the infiltrated material were exceptionally complex; parts of the SiB_6 and AlB_{12} diffraction patterns could be seen reflecting the complexity of the microstructure as evidenced by electron probe microanalysis. One interesting point was the very high degree of preferred orientation of the (111) silicon reflection, developed during the solidification of the liquid silicon infiltrant.

4. B_4C (NC)-Si, B_4C (BPI)-Si, and B_4C/boron-Si mixtures. The B_4C (NC)-Si compact was completely infiltrated, and its microstructure is shown in Figure 2a.

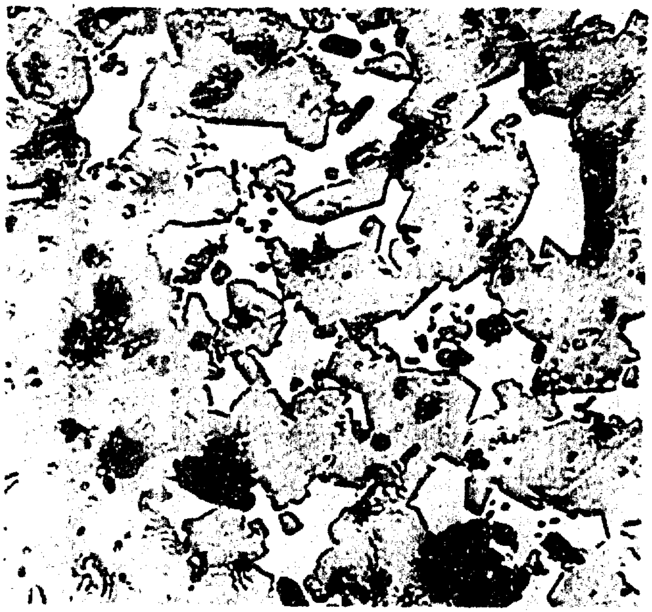
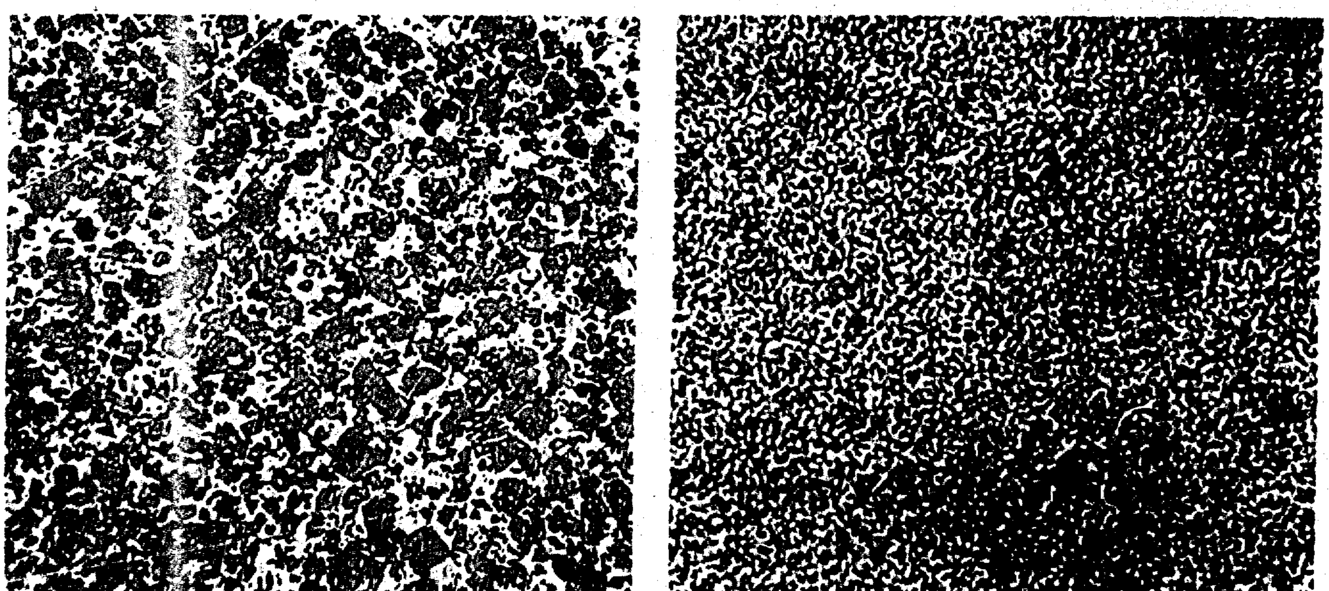


Figure 1. Aluminum boride (-320 mesh) infiltrated with silicon. Mag. 500X



a. NC (-320 mesh); Mag. 100X

b. BPI; Mag. 500X

19-066-1249/AMC-71

Figure 2. Boron carbide infiltrated with silicon

The B_4C (BPI)-Si compact had a sharp demarcation line about 5 mm in from the edge of the 25.4-mm-diameter compact, the internal part of the compact being uninfiltrated; the penetrated part of the compact had a fully infiltrated microstructure appearance (see Figure 2b). This difference is probably a pore size effect and is discussed more fully in the section on Mechanical Property Results.

The B_4C/boron-Si mixtures showed areas of poor boron/B_4C powder mixing but otherwise their microstructures were similar to the 100% B_4C microstructure. Boron, a hard material (Knoop hardness[5] about 2500 $kg.mm^{-2}$ for a 100-g load), was added as a "sink" for silicon with possible SiB_6 formation and a reduction of area of the relatively low hardness silicon infiltrant. The 80% B_4C (NC) 20% boron was cracked but single specimens from the B_4C (NC)-Si and the 90% B_4C (NC)/10% boron-Si materials were mechanically tested. These results together with densities are given in Table II. The combined mechanical property data for silicon and aluminum infiltrations are discussed later.

Single Infiltration Experiments with Aluminum

1. B_4C (NC)-Al. No evidence of infiltration under the conditions employed.

2. SiB_6-Al. Good infiltration obtained to yield a compact of density of 2.47 g/cc (a 60 w/o SiB_6/40 w/o Al infiltrated material would have a density of 2.54 g/cc). Hardness values were measured but it was difficult to place the indenter on single component areas. The highest values obtained on the grey areas of the microstructure (see Figure 3) were about 2300 $kg.mm^{-2}$, similar to the value for SiB_6.

3. Boron-Al. Good infiltration was obtained to yield a fine textured microstructure (see Figure 4); some residual porosity was present as a measured density of 2.32 g/cc was determined. Some limited mechanical property data is discussed in the next section.

4. AlB_{12}-Al and AlB_{12}/boron-Al mixtures. The three materials AlB_{12}, 80% AlB_{12}/20% boron and 60% AlB_{12}/40% boron appeared to be completely infiltrated as judged by their microstructures. Mechanical property data were obtained on the AlB_{12}-Al and 60% AlB_{12}/40% boron-Al materials.

The microstructure of the boron additive materials showed gross regions of nonuniform mixing of the boron and AlB_{12} powders. The microstructure of the AlB_{12}-Al material was of interest and Figure 5a shows the continuous interface between the infiltrated aluminum, aluminum boride, and the excess essentially cast aluminum (but with grain boundary precipitates) not taken up during infiltration; Figure 5b shows part of this interfacial zone at a higher magnification and Figure 5c is a micrograph of zone C, the infiltrated AlB_{12} matrix.

X-ray diffraction data covering the zones A, B, and C for the AlB_{12}-Al system showed some characteristics demonstrated by all the Al/B-Al systems investigated. The AlB_{12} diffraction lines were still present but the pattern was more complex than the AlB_{12} powder pattern (possible AlB_{10} phases). There was no evidence of AlB_2 formation (a compound with relatively low hardness about 1000 Knoop), reported to form between its elements at about 1100 C;[5] as with the silicon infiltrated materials there was a very high degree of preferred orientation on the (111) aluminum reflection at 2.338 Å.

Table II. MECHANICAL PROPERTIES OF INFILTRATED CERAMICS
(0.12 in. × 0.12 in. × 0.8 in. bend spec.)

System	Density g/cc	Bend Strengths × 10^3 psi	Bend Strengths $MN.m^{-2}$	Elastic Modulus × 10^6 psi	Elastic Modulus $GN.m^{-2}$
B_4C-Si	2.48	26.1	180	49.7	340
90 w/o B_4C/10 w/o B-Si	2.47	12.3	85	38.9	270
AlB_{12}-Al	2.59	47.9	330	32.3	220
60 w/o AlB_{12}/40 w/o B-Al	2.47	30.0	207	19.5	130
B-Al	2.32	17.5	121	16.7	120

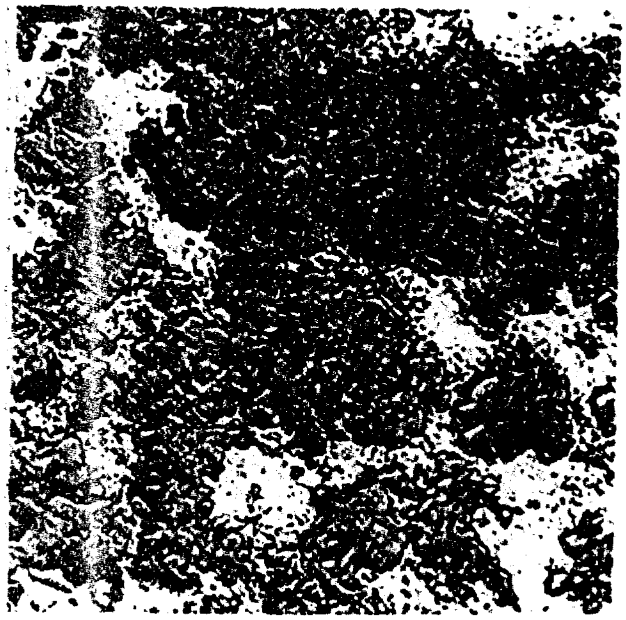


Figure 3. Silicon hexaboride (-320 mesh) infiltrated with aluminum. Mag. 500X

19-066-1032/AMC-72

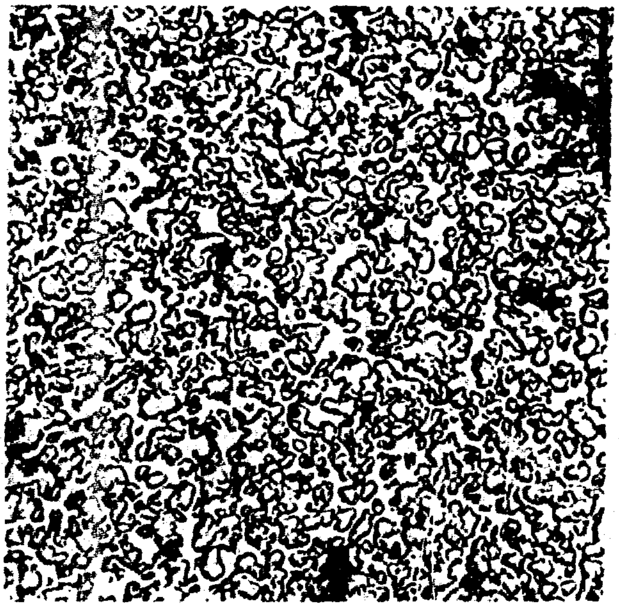


Figure 4. Boron powder compact infiltrated with aluminum. Mag. 500X

19-066-1250/AMC-71

Electron probe microanalysis studies on this material yielded the following information. In Figure 5a, zone A is pure aluminum with a fine grain boundary precipitate of probably Al_2O_3. In Figure 5b the interfacial zone B is 100% aluminum and the large particles in the interfacial zone are AlB_{12}. In Figure 5c large grey grains were identified as being of both AlB_{12} and AlB_{10} types while the interconnecting matrix was 100% Al.

MECHANICAL PROPERTY RESULTS AND DISCUSSIONS OF SINGLE INFILTRATION STUDIES

The mechanical properties of some of the more promising infiltrated ceramics are given in Table II, and the actual bend stress/strain curves (4-point testing) are shown in Figure 6.

A conclusive difference in behavior can be seen between the silicon-infiltrated ceramics (curves 1 and 2 in Figure 6), which deform elastically to fracture, and the aluminum-infiltrated materials (curves 3 to 5), which have a relatively small elastic region up to approximately 7000 psi (48 $MN.m^{-2}$), when plastic deformation and yield occur until final fracture at relatively large strain values (110 μm, which is approximately ten times the strain levels of the B_4C-Si brittle systems). This difference in fracture behavior is clearly seen in Figures 7 and 8, scanning electron micrographs of fracture surfaces generated during mechanical testing of B_4C-Si and AlB_{12}-Al; the brittle fracture characteristics of the B_4C-Si fracture face contrast with the ductile tearing of the aluminum around the AlB_{12} grains in Figure 8. The B_4C-Si properties are in the same region as properties obtained with commercial modified boron carbides.

Three bend bars were machined from a 2-inch-diameter AlB_{12}-Al infiltrated compact. Results show that the modulus of rupture (MOR), 45,000 to 48,000 psi, was similar to that for the smaller specimens but the elastic modulus, approximately 25 × 10^6 psi, was somewhat lower than that determined for the smaller specimens. Elastic moduli were also determined by a sonic technique reported by Hasselman[6] and values were found to be approximately equal to those determined mechanically. The AlB_{12}-Al result is particularly interesting and a comparison with some high strength aluminum alloys is given in Table III.[7]

Based on the law of mixtures and taking a value of 57 × 10^6 psi for the elastic modulus of AlB_{12}, a value of 32 × 10^6 psi would correspond to a 50:50 AlB_{12}/Al composite. The high MOR value for the AlB_{12}-Al system coupled with the hardness values of approximately 2500 $kg.mm^{-2}$ for the AlB_{12} component, coupled with its low density (2.59 g/cc), make this an interesting armor material.

It is probable that with a suitable heat-treatable casting alloy infiltrant, considerable increases in yield stress could be achieved in the AlB_{12}-Al system, together with improvements in the MOR value.

This work on single systems represents a preliminary survey of some potential metal-infiltrated ceramic systems. Much remains to be done to optimize processing conditions, compositions, particle size/pore size characteristics and infiltrant compositions and temperatures. The two variables of immediate importance are those represented in the Washburn-Rideal equation (see the following section), namely pore size (controlled by particle size, compaction pressure, pre-sintering,

Aluminum zone A

Aluminum boride-aluminum interfacial zone B

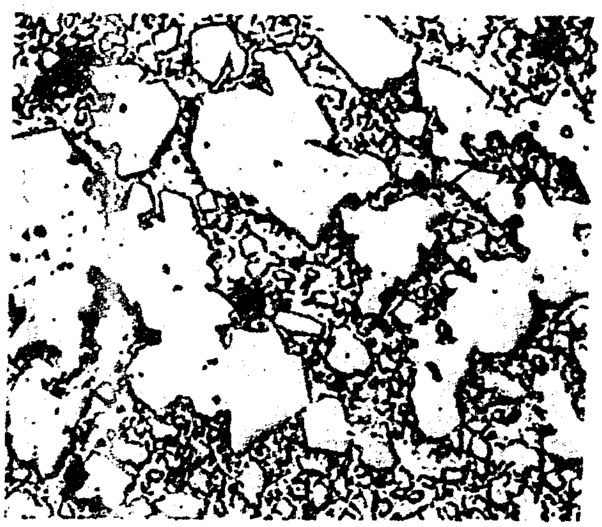
Aluminum boride matrix infiltrated with aluminum zone C

a. Mag. 100X

Aluminum boride-aluminum interfacial zone B

Aluminum boride matrix infiltrated with aluminum zone C

b. Mag. 500X



c. Mag. 500X

Figure 5. Aluminum boride (-320 mesh) infiltrated with aluminum

or the deliberate addition of porosifiers) and viscosity of the infiltrant (controlled by temperature and composition of the liquid metal). The work is directed to those systems that are likely to be suitable component parts for dual infiltration systems.

DISCUSSION AND DUAL INFILTRATION RESULTS

The theory of interfacial conditions in solid/liquid/vapor systems has been given by Davies and Rideal[8] and using their thesis it is possible to calculate the changes in surface energy on liquid metal infiltration into a porous ceramic, assuming a stable porous ceramic structure.

The infiltrated material is stable with respect to the porous ceramic preform and the liquid metal infiltrant if

$$(S_L - A_2)\,\gamma_{LV} + A_1\,(\gamma_{SV} - \gamma_{SL}) > 0 \qquad (1)$$

where S_L = surface area of liquid before infiltration

A_1 = internal surface area of ceramic porosity prior to infiltration

A_2 = surface area of external pore entrances

$$\left.\begin{array}{l} \gamma_{LV} = \text{surface energy liquid to vapor} \\ \gamma_{SV} = \text{surface energy solid to vapor} \\ \gamma_{SL} = \text{surface energy solid to liquid} \end{array}\right\} \text{in the infiltration environment}$$

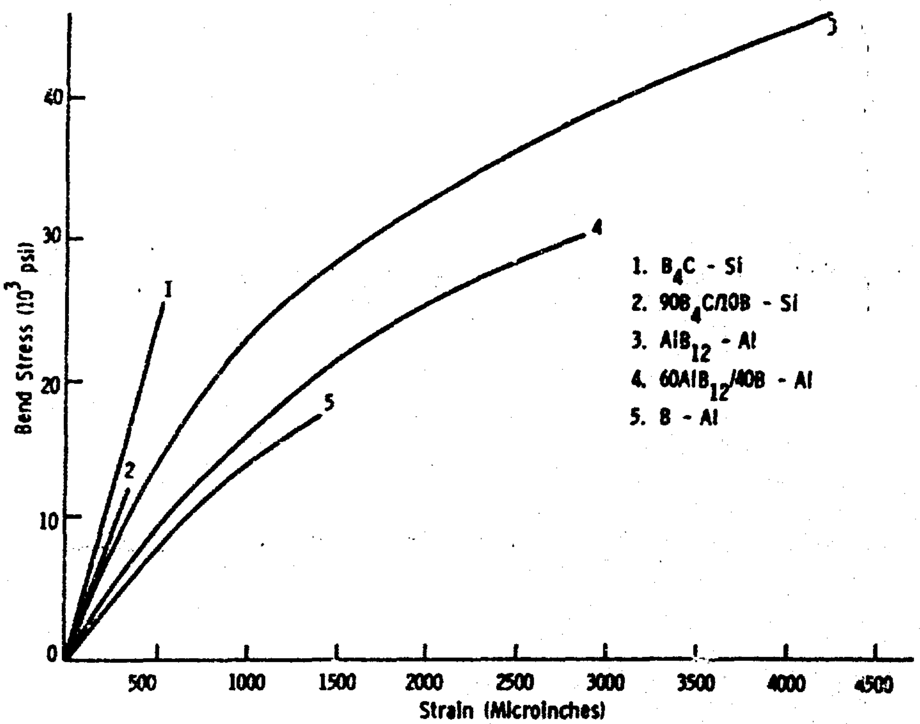


Figure 6. Bend stress versus strain for infiltrated ceramics

19-066-1255/AMC-71

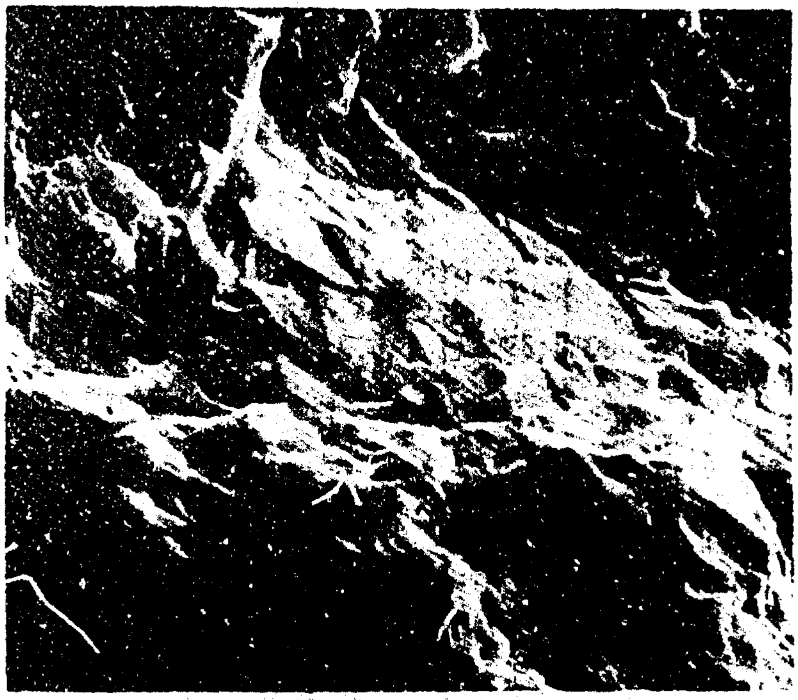


Figure 7. Fracture surface of -320 mesh B_4C infiltrated with Si. Mag. 850X

19-066-1252/AMC-71

Figure 8. Fracture surface of -320 mesh AlB_{12} infiltrated with Al. Mag. 450X

19-066-1252/AMC-71

Table III. MECHANICAL PROPERTIES OF SOME COMMERCIAL Al ALLOYS[7]

Material	Proof Stress		UTS		Elongation	Elastic Modulus	
	psi	$MN.m^{-2}$	psi	$MN.m^{-2}$	%	$\times 10^6$ psi	$GN.m^{-2}$
99.9% Al, sand cast	5,000 (yield)	35	10,000	70	11	10	69
99.9% Al, sand cast and cold worked	22,000	150	24,000	165	~5	10	69
Al-12% Si, cast and heat treated	10,000	70	26,000	180	8	-	-
Al-5.5% Zn-3.0% Mg-1.5% Cu-0.7% Mn, cast and heat treated	56,000	390	67,000	460	12	-	-
AlB_{12}-Al (approximately 65% AlB_{12} - this work)	10,000 (yield)	70	48,000 (MOR)	330	~5	32	220

Since $A_1 >> S_L$ and A_2, Eq. 1 reduces to $A_1 (\gamma_{SV}-\gamma_{SL}) > 0$ (2)

The infiltration rate R is given by the Washburn-Rideal[8] equation as

$$R \propto \frac{r}{\eta l} (\gamma_{SV}-\gamma_{SL}) \quad (3)$$

where r = mean radius of pores

l = linear distance penetrated by the infiltrant

η = viscosity of liquid metal at the infiltration temperature

Thus an equivalence exists such that a decrease in pore size favors the process energetically but actually leads to a reduction in infiltration rate. (The decrease in pore size, at the same total volume fraction porosity, is associated with an increase in internal solid surface area.)

Williams,[9] however, lists factors that limit the application of the above ideas to very small pore sizes. These are:

(a) the increase in the retarding effect to the infiltrant due to internal surface roughness with decreasing pore size;

(b) a greater tendency for film formation and gas entrapment at finer pore sizes; and

(c) where chemical compatibility at liquid infiltration temperatures causes problems, the faster infiltration permitted by larger pore sizes would be advantageous.

In the absence of data on the variation of viscosity of liquid aluminum and liquid silicon with temperature the validity of the Wasburn-Rideal relationship (Equation 3) could not be tested. However, for the infiltration of liquid silicon into two types of boron carbide, the variation of infiltration rate with

pore radius was observed. Under identical conditions, a -320 mesh (44 μm), 25.4-mm-diameter pressed B_4C compact was totally infiltrated (typical microstructure, Figure 2a) whereas a similarly pressed Boride Products B_4C powder (particle size 10 μm) had a sharp demarcation line about 5 mm in from the edge of the 25.4-mm-diameter compact, the internal part of the compact being uninfiltrated. That part of the Boride Products B_4C compact that had been infiltrated had zero apparent porosity (see Figure 2b).

Gradient Ductile/Brittle Material Based on a Single Component Ceramic - The Effect of Particle Size

The observation of the different infiltration rates in identical materials differing only in their pore structures leads to the following interesting hypothesis:

> In a sandwich one-component porous pressed ceramic, where the porous front face consists of a different (larger) particle size and pore structure than the rear half of the sandwich, it should be possible, by controlling infiltration rates, to completely penetrate the large porosity of the front face with a high temperature brittle infiltrant while achieving minimal or negligible penetration of the fine porosity rear face. In a second stage to such a process, a lower melting point ductile infiltrant could then be infiltrated over a longer period of time into the previously unpenetrated fine porosity of the rear face without influencing the previously infiltrated brittle front half of the sandwich.

<u>Brittle Component</u> Large pore size, large particle size and high temperature brittle infiltrant	FRONT FACE
<u>Ductile Component</u> Fine pore size, fine particle size and lower temperature ductile infiltrant	REAR FACE

Such a system would approach the criteria suggested by Wilkins[2] for an improved ceramic armor with some ductility in the rear face.

A single unsuccessful attempt was made to produce such a system based on alpha-AlB_{12} since it was known that this material could be infiltrated by both silicon and aluminum. AlB_{12} powder was hand sieved into the -400 mesh and +400 mesh components and the die was charged with a layer of each sieve fraction and then cold pressed at 17,700 psi (123 $MN.m^{-2}$). Liquid silicon appeared to infiltrate preferentially into the +400 mesh side of the compact and in the second infiltration step the remainder of the compact was totally infiltrated with aluminum. Examination by electron probe analysis surprisingly showed a very low level of silicon (about 1% to 2%) in both the +400 mesh and -400 mesh components with approximately a 0.4% higher level in the +400 mesh region. The main phases seen were alpha-AlB_{12} and aluminum with some Al_2O_3 elongated inclusions. It is suspected that the differences in particle/pore sizes for the two halves of the component were too small, but it is felt that the principle could be made to work. It is probable that the dissimilar material gradient system will be more successful.

Gradient Ductile/Brittle "Ceramic" Based on a Two-Component Ceramic - The Effect of Wettability

From the work on the infiltration of single component systems it has been found that not all potential armor ceramics are wet and penetrated by liquid silicon and liquid aluminum under the conditions investigated. For example, SiB_6 is wet by aluminum but not by silicon (apart from an unidentified 150 μm skin effect) and B_4C is wet by silicon but not by aluminum. This leads to the following interesting hypothesis:

> It should be possible to fabricate a sandwich two-component pressed porous ceramic where the porous front face consists of a ceramic X penetrated by a liquid metal A at a temperature T_1 and the rear face consists of a ceramic Y which is not penetrated by A at a temperature T_1. The liquid metal A on solidifying yields a brittle connecting phase around a hard brittle phase X. In a second stage to this process a ductile metal infiltrant B is selected that will wet and penetrate the previously unpenetrated rear face Y at a temperature T_2 (where $T_1 > T_2$) without influencing the previously penetrated fully dense brittle phases X + A.

<u>Wholly Brittle Component</u> - 1st stage	FRONT FACE
Ceramic X, wet and penetrated by liquid metal A at temperature T_1 Ceramic X not affected by liquid metal B	
<u>Brittle/Ductile Component</u> - 2nd stage	REAR FACE
Ceramic Y, wet and penetrated by liquid metal B at temperature T_2 Ceramic Y not penetrated by liquid metal A Temperature $T_1 > T_2$	

Such a system would also approach the criteria suggested by Wilkins for an improved ceramic armor with some ductility in the rear face. The relative thicknesses of X and Y could, of course, be varied and a graded interface between the two parts could also be evolved.

The B_4C-Si/SiB_6-Al systems mentioned above have been investigated from this standpoint and an early result looks interesting. A 10-mm-deep layer of SiB_6 powder was loaded on top of a 10-mm-deep layer of -320 mesh B_4C powder in a double acting 25.4-mm-diameter die set and cold pressed at 17,700 psi (123 $MN.m^{-2}$) to yield a single compact. Liquid silicon was infiltrated into the B_4C part of the compact but the liquid silicon did not wet the SiB_6 part of the compact: the previously described experimental procedures were used. In a second stage, liquid aluminum was successfully infiltrated into the SiB_6 part by the methods previously described for aluminum infiltration. A fractograph of the cross section of this system is shown in Figure 9.

Although mechanical properties have not as yet been determined for this system, one can speculate that the mechanical behavior might be somewhere between that of the AlB_{12}/Al system exhibiting ductility and that of the B_4C/Si showing brittle behavior.

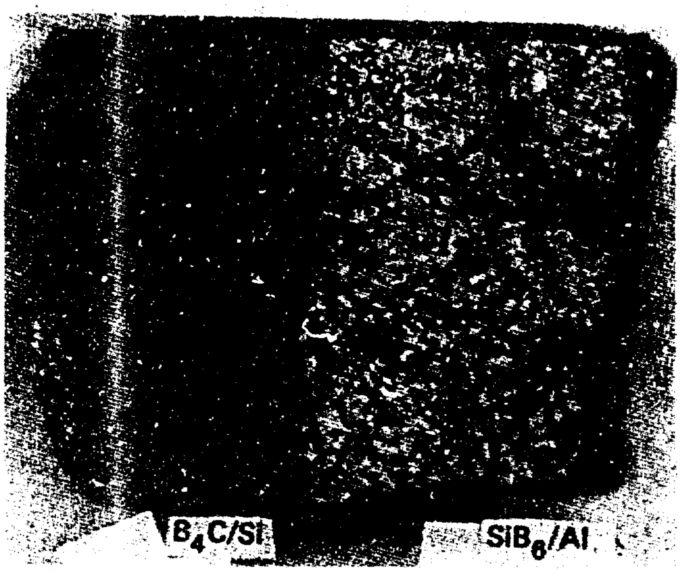


Figure 9. Fractograph of a B_4C/Si - SiB_6/Al dual-infiltrated system. Mag. 10X

SUMMARY

Liquid metal infiltration of various porous ceramics offers a potentially inexpensive method of fabricating armor materials as demonstrated commercially in the "modified B_4C" material. It has been shown that other systems based on a ductile metal infiltrant (aluminum) can also be fabricated with interesting mechanical properties such as limited ductility, relatively high elastic modulus and high strength (AlB_{12}-Al system). Limited mechanical property and microstructural data and comments on fabricability of various aluminum- and silicon-infiltrated armor systems are presented.

The concept of a gradient armor system with a hard brittle front face and a ductile rear face has been investigated from two standpoints. In one experiment it was not possible to fabricate a gradient system by dual infiltration of a one-component system by pore size control; although initial attempts were unsuccessful it is felt that such a system might still be made to work.

In other experiments it has been found feasible to fabricate a gradient armor system based on the B_4C/Si-SiB_6/Al system consisting of a hard brittle front face and a hard rear face but with an interconnecting ductile network of aluminum. However, a considerable amount of work needs to be done to optimize such a system from the standpoint of materials and processing. Such systems also require scale-up and ballistic assessment but the inherent properties of the individual components could go some way toward the requirements for an improved armor system advocated by Wilkins.[2]

ACKNOWLEDGMENTS

Appreciation is extended to Mr. I. Berman (Materials Development and Engineering Laboratory, AMMRC), Dr. R. N. Katz (Ceramics Research Division, AMMRC), and Dr. D. J. Godfrey (Admiralty Materials Laboratory, U. K.) for initiating arrangements, through TTCP, Subpanel P-2, for the scientific exchange, and to the Ministry of Defence (Navy Department, U. K.) through the Director of the Admiralty Materials Laboratory for permitting Dr. Lindley to participate in this program.

LITERATURE CITED

1. WONG, A. K., and BERMAN, I. *Lightweight Ceramic Armor - A Review.* Army Materials and Mechanics Research Center, AMMRC MS 71-1, April 1971.

2. WILKINS, M. L., CLINE, C. F., and HONODEL, C. A. *Fourth Progress Report of the Light Armor Program.* Lawrence Radiation Laboratory, Livermore, California, Report UCRL - 50694, 1969.

3. STIGLICH, J. J., RANKIN, D. T., PETRAK, D. R., and RUH, R. *Characterization of Hot-Pressed Al_2O_3 with a Mo-Dispersed Phase.* Army Materials and Mechanics Research Center, AMMRC TR 70-35, December 1970.

4. GOETZEL, C. G., and ADAMEC, J. B. *Infiltration of Cermets for Improved Toughness.* Metals Progress, v. 70, no. 6, 1956, p. 101.

5. KOHN, J. A., NYE, W. F., and GAULE, G. K. *Boron - Synthesis, Structure and Properties.* Plenum Press, 1960.

6. HASSELMAN, D. P. H. *Tables for the Computation of the Shear Modulus and Young's Modulus of Elasticity from the Resonant Frequencies of Rectangular Prisms.* Research & Development Report, Carborundum Co., Niagara Falls, New York, 1961.

7. SMITHELLS, L. J. *Metals Reference Book.* Published by Butterworths, 1967.

8. DAVIES, J. T., and RIDEAL, K. E. *Interfacial Phenomena.* Academic Press, 1961.

9. WILLIAMS, L. S. *Infiltrated Oxide Ceramics.* Chapter 11 in "Special Ceramic," 1964, Academic Press, 1965.